

## A TECHNIQUE TO MEASURE THE THERMAL DIFFUSIVITY OF HIGH $T_c$ SUPERCONDUCTORS

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High temperature superconducting electrical current leads and ground straps will be used in cryogenic coolers in future NASA Goddard Space Flight Center missions. These superconducting straps will be long, thin leads with a typical diameter of 0.2 cm. A longitudinal method is being developed to measure the thermal diffusivity of candidate materials for this application. This technique will use a peltier junction to supply an oscillatory heat wave into one end of a specimen and will use low mass thermocouples to follow the heat wave along the specimen. The thermal diffusivity will be calculated using both the exponential decay of the heat wave and the phase shift of the wave. Measurements will be done in a cryostat between 10 K and room temperature.

### INTRODUCTION

A program has been initiated in the Materials Branch at the Goddard Space Flight Center to develop methods for characterizing candidate high temperature superconducting (HTSC) material for possible space flight use. Part of this program has been directed at measuring various physical quantities of HTSC electrical current leads that may be used in cryogenic coolers. These current leads will typically be between 0.1 and 0.2 cm in diameter. The advantages of using current leads constructed from HTSC material over conventional current leads are higher electrical conductivity and lower thermal conductivity. The disadvantages of using HTSC material are its mechanical properties. Since all HTSC materials are ceramics, they have a low ductility and have a widely varying strength due to inherent defects typical of ceramics.

In this paper, preliminary measurements of the thermal diffusivity,  $D$ , of a  $YBa_2Cu_3O_{7-x}$  specimen are reported. These thermal diffusivity measurements will be used along with thermal conductivity and specific heat measurements, to be made using the same apparatus, to characterize the thermal properties of HTSC specimens.

The technique used to measure thermal diffusivity is an implementation of Angstrom's temperature wave method.<sup>1</sup> A heater is attached to one end of a specimen rod to send a periodic heat wave down the rod. Two thermometers are mounted along the specimen to follow the wave. The phase difference between the temperature oscillations at the two thermometers and the ratio of the amplitudes of these oscillations are used to calculate  $D$ .

### SPECIMEN DESCRIPTION

The HTSC specimen used for these experiments is manufactured by Argonne National Laboratory. The x-ray diffraction pattern for the specimen shows it to be single phase  $YBa_2Cu_3O_{7-x}$  with trace amounts of barium oxide and other impurities. AC susceptibility measurements (Figure 1) done at 100 kHz, in the absence of an external magnetic field, show the specimen to have a critical temperature of 90 K and a transition region of about 2 K. The specimen has a cylindrical shape with a diameter of 0.188 cm, a length of 2.556 cm, and a density of 5.93 gm/cm<sup>3</sup>.

### EXPERIMENTAL METHOD

In this implementation of the temperature wave method, a peltier junction is used to generate a periodic heat wave and two chromel-alumel thermocouples made of 0.0076 cm diameter wire are used to measure the temperature along the specimen rod (Figure 2). One thermocouple is connected to the specimen 0.487 cm from the peltier junction, and the other is 1.799 cm from the peltier junction. The peltier junction is mounted on a block of aluminum that is used to control the absolute temperature at which a measurement is made. Running through the aluminum block are

several copper tubes through which liquid nitrogen is allowed to flow to control the temperature of the peltier junction and specimen rod. Presently, measurements are made in vacuum ( $10^{-5}$  torr) between 150 and 300 K. The use of a cryostat in the future will allow measurements to be made between 10 and 300 K. The peltier junction is connected to the block of aluminum and the specimen rod using STYCAST 2850FT epoxy manufactured by Emerson & Cuming. The thermocouples are also connected to the specimen rod using STYCAST 2850FT.

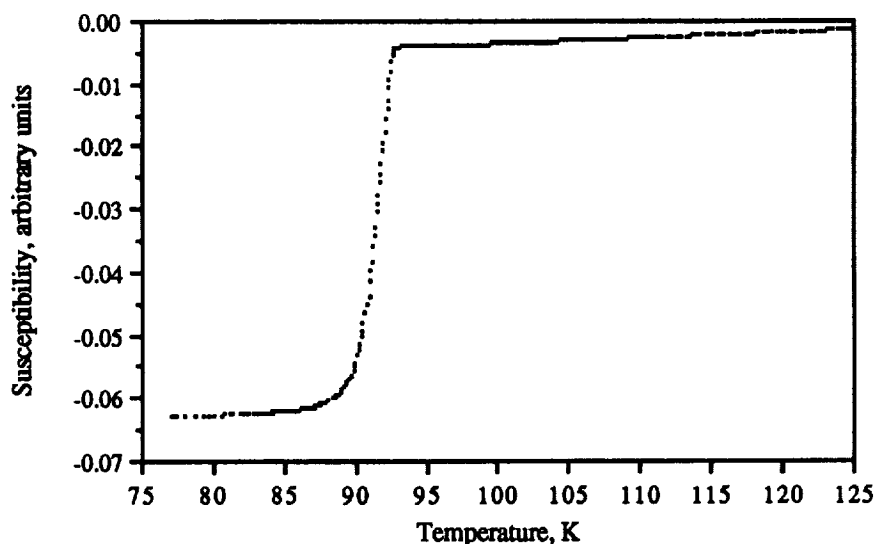


Figure 1. AC susceptibility of the  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  specimen used in these experiments.

A Hewlett-Packard 3314A function generator connected to a Kepco BOP 50-2M amplifier are used to produce an oscillating current through the peltier junction. The function generator is capable of generating a sinusoidal signal with a frequency as low as .001 Hz. A Fluke 8505A digital multimeter and a Keithley 196 system digital multimeter are used to measure the voltage across each thermocouple. Both multimeters have a resolution of 100 nV (this is about 0.0025 K for chromel-alumel thermocouples). A Fluke 1722A instrument controller is used to record the measurements from the multimeters.

By controlling the amount of liquid nitrogen that flows through the copper tubes mentioned previously, the absolute temperature at which a diffusivity measurement is made can be set. Once the temperature of the aluminum block and test specimen have reached equilibrium, a periodic current is supplied to the peltier junction to generate a thermal wave through the specimen rod. When the temperature oscillations at each thermocouple reach steady state, a BASIC program is initiated on the Fluke 1722A instrument controller to read and record the voltage across each thermocouple. For these experiments the frequency of oscillation of the thermal wave ranges between .01 and .05 Hz, so the voltage across each thermocouple is sampled once a second giving between 20 to 100 measurements per cycle.

## DATA ANALYSIS

To calculate the thermal diffusivity from the voltage measurements mentioned previously, a curve fitting routine is used to determine the magnitude and phase of the temperature oscillations at each thermocouple. These values are then used in the following equation to calculate diffusivity,

$$D = (\pi f L^2) / [\phi \ln(a_0/a_1)] \quad (1)$$

where  $f$  is the frequency of oscillation,  $L$  is the distance between the thermocouples,  $\phi$  is phase different between the thermocouples,  $a_0$  is the amplitude of the temperature oscillation at the thermocouple closest to the peltier junction,

and  $a_1$  is the amplitude of the temperature oscillation at the second thermocouple. This equation assumes that the amplitude of the heat wave has decayed to a negligible value at the end of the specimen farthest from the peltier junction.

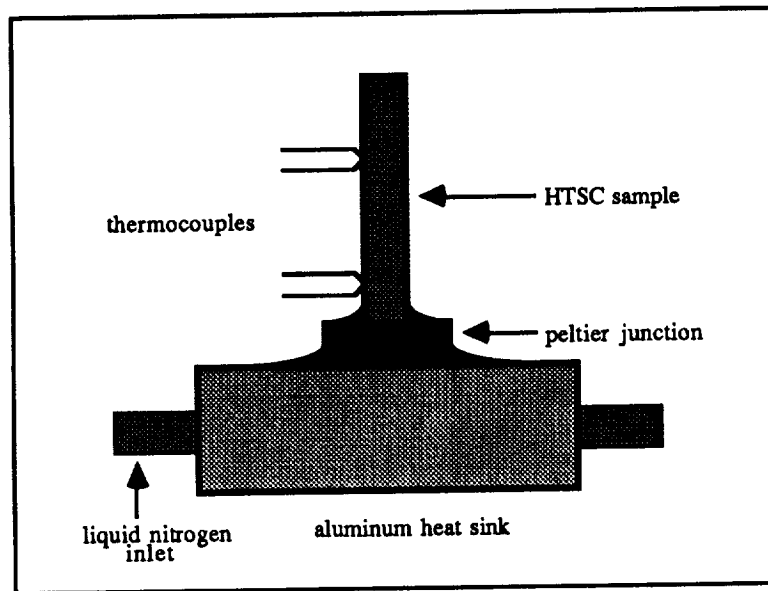


Figure 2. Basic apparatus for measuring thermal diffusivity.

For negligible lateral heat losses to the surrounding environment or thermocouples the phase difference between the thermocouples is approximately equal to the logarithm of the amplitude ratio, and the following equation can be used to calculate diffusivity,

$$D = (\pi f L^2) / (\phi^2) = (\pi f L^2) / [\ln(a_0/a_1)]^2. \quad (2)$$

In these experiments, the value of diffusivity calculated using only the phase difference is always about 10% larger than the values calculated using the amplitude ratio, indicating some lateral heat loss. Modified diffusivity calculations which account for losses due to the thermocouples only predict the value calculated from the phase difference to be about 2% larger than the value from the amplitude ratio. This 10% difference is characteristic of a lateral heat loss, which presently has not been identified. Radiative coupling with the surrounding environment could be the cause of this heat loss. During these experiments, measurements below room temperature always had a temperature gradient across the specimen, which can be entirely accounted for by radiative coupling calculations.

Two alternate equations, which account for the reflection of the heat wave at the end of the rod farthest from the peltier junction, can be used to calculate the diffusivity. These equations are,

$$(a_0/a_1) = \tan^{-1}[\tan(kx_0)\tanh(kx_0)] - \tan^{-1}[\tan(kx_1)\tanh(kx_1)] \text{ and} \quad (3)$$

$$\phi = \sqrt{[\cosh(2kx_0) + \cos(2kx_0)] / [\cosh(2kx_1) + \cos(2kx_1)]} \quad (4)$$

where  $k = \sqrt{\pi f / D}$ , and the lengths  $x_0$ ,  $x_1$  are measured from the free end of the rod to the thermocouples mounted closest and farthest respectively from the peltier junction.<sup>2</sup> The diffusivity values calculated from equation (2) are always within two percent or better of the values calculated with equations (3) and (4), indicating that the amplitude of the thermal wave at the end of the rod farthest from the peltier junction is negligible for these calculations.

The diffusivity of a cylindrically shaped fused  $\text{SiO}_2$  specimen (Corning 7940) with a diameter of 0.404 cm and a length of 11.9 cm was measured using this technique to test the accuracy of the technique. The measured values (Figure 3) agree to within  $\pm 6\%$  of the values reported by Luikov, Vasiliev, and Shashkov.<sup>3</sup> Most of the uncertainty in these measurements is due to thermal noise associated with the thermocouple contacts. The amplitude of the temperature wave at the thermocouple closest to the peltier junction is typically 0.6 K (1.2 K peak-to-peak), and the amplitude at the other is typically 0.03 K, while the standard deviation of the noise is 0.015 K. When this noise is simulated using a computer program a  $\pm 5\%$  uncertainty in the generated diffusivity values is observed, which is in good agreement with the measured values.

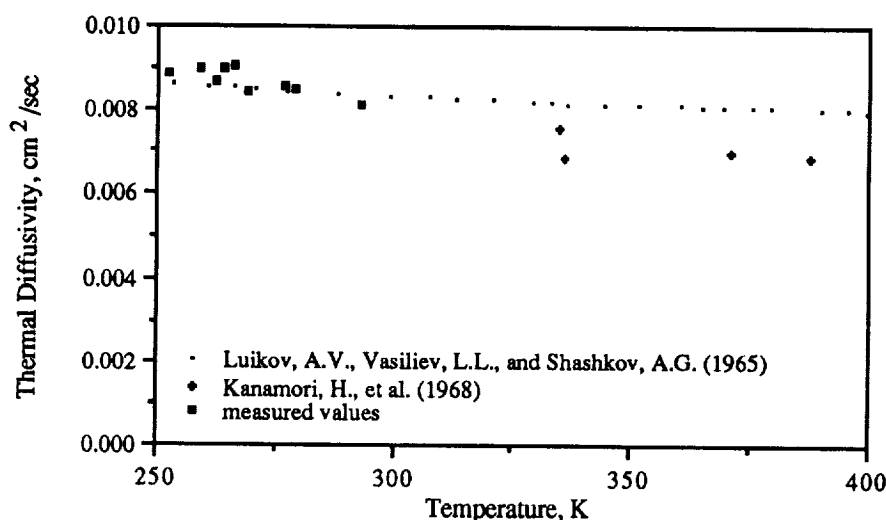


Figure 3. Thermal diffusivity of fused  $\text{SiO}_2$ .

## RESULTS

The thermal diffusivity of the  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  specimen, calculated using equation (1), is shown in Figure 4. All measured values of diffusivity are within  $\pm 4\%$  of a second order polynomial fit to the data. Again, the amplitude of the temperature wave at the thermocouple closest to the peltier junction is typically 0.6 K, and the amplitude at the other is typically 0.03 K, but the standard deviation of the noise is only 0.012 K. When this noise is simulated using a computer program a  $\pm 4\%$  uncertainty in the generated diffusivity values is observed, which is in good agreement with the measured values as compared to the polynomial fit.

## CONCLUSION

From the measured values of thermal diffusivity for fused  $\text{SiO}_2$ , as compared to other reported values, it is concluded that the accuracy of this technique is about  $\pm 6\%$ . Further, the agreement in uncertainty between the measured values and the computer simulated values indicate that most of the error is due to noise associated with the thermocouples. There may also be a systematic error of about 2% due the distance measurement between the thermocouples.

To improve the accuracy of this technique, several changes will be made in the future. The first will be the use of a cryostat, which will decrease the amount of radiative coupling of the specimen with the surrounding environment and will allow for measurements down to 10 K with better temperature control. The thermocouples to be used in future measurements will have a diameter of 0.0025 cm, which will decrease heat losses to the thermocouples and will increase the accuracy of the distance measurement between the thermocouples. These thermocouples will also be connected in series with junction thermocouples (that will be connected to the block of aluminum) to eliminate any DC voltage across the thermocouples due to the absolute temperature of a measurement. This voltage is typically

100 times larger than the magnitude of the voltage oscillations due to the heat wave. Eliminating this offset will increase the accuracy of the temperature measurements by allowing the use of voltage amplifiers and filters.

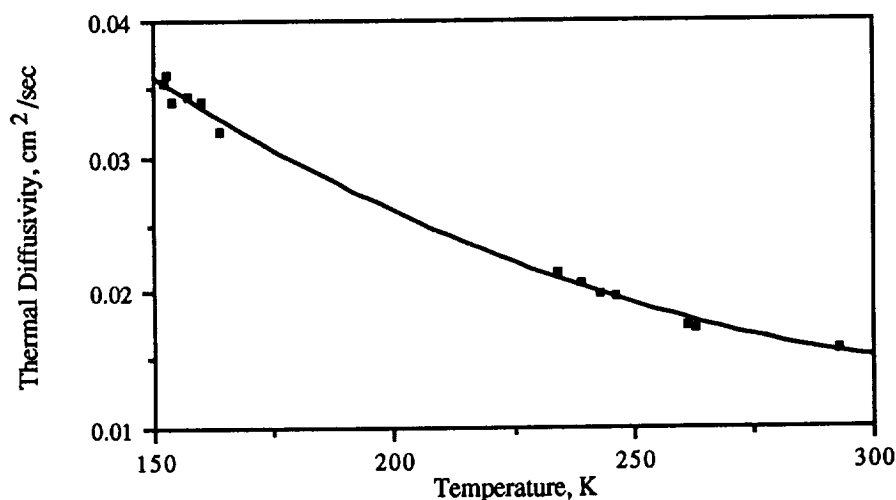


Figure 4. Thermal diffusivity of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  specimen.

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#### REFERENCES

1. Touloukian, Y.S., Powell, R.W., Ho, C.Y., and Nicolaou, M.C., Thermal Diffusivity -- Vol. 10 of Thermophysical Properties of Matter -- The TPRC Data Series, pp. 28a-37a, IFI/Plenum Data Corp., New York, 1973.
2. Howling, D.H., Mendoza, E., and Zimmerman, J.E., "Preliminary Experiments on the Temperature-Wave Method of Measuring Specific Heats of Metals at Low Temperatures", A229, pp. 86-109, 1955.
3. Touloukian, Y.S., Powell, R.W., Ho, C.Y., and Nicolaou, M.C., Thermal Diffusivity -- Vol. 10 of Thermophysical Properties of Matter -- The TPRC Data Series, pp. 399-400, IFI/Plenum Data Corp., New York, 1973.

